

~~SECRET~~*To - Ballou, Tarkenton, Hyarogun, Gen.*

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*Rec'd ED
6/11/59
1600*

June 8, 1959

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Dear Sir:

This letter report describes the research performed under
Task Order No. C during the month of April, 1959.

Eight runs were made during April. These included the
following:

- (1) Four small-scale runs, designed to obtain additional information on the effect of the mode of catalyst addition on the total generation time.
 - (2) Two 1/10-scale runs at initial temperatures of 33 and 34 F, designed to determine the effect of low temperatures on the total generation time.
 - (3) Two additional 1/10-scale runs at initial temperatures of 37 and 42 F, designed to explore how the limited solubility of the catalyst at low temperatures might impose a limitation on the amount that could be used to attain the desired reaction time.
- Also, a limited study of the catalyst solubility is being made to resolve apparent errors in the published solubility data.

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The small-scale Runs 4, 5, 6, and 7 were carried out in a 5 gallon distilled-water bottle about 10-1/2 inches in diameter. The volume of the reaction pool was 7-1/2 liters, with a depth of approximately 5-3/4 inches. The NaBH_4 concentration was 0.56 molar and the catalyst concentration in the whole reaction solution was 0.005 molar. The hydrogen generated was bubbled through water in order to facilitate detecting the end of the reaction.

In Runs 4 and 6, the catalyst solution was all added at the center of the reaction pool. In Run 5, the catalyst solution was distributed manually to some degree over the surface of the NaBH_4 solution, but completely uniform distribution was not possible. In Run 7, the NaBH_4 solution was manually stirred during addition of the catalyst solution and for a 30-second period thereafter. The results obtained are as follows:

Run No.	Initial Temperature, T_0 , F	ΔT , F	Total Generation Time, min	Mode of Catalyst-Solution Addition
4	68	50	42	Center pour
5	64	48	51	Distributed over surface of NaBH_4 solution
6	70	50	39	Center pour
7	71	51	25	NaBH_4 solution stirred

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Comparison of the results of Runs 4 and 6 with those of Run 7 shows that the reaction time was reduced markedly in Run 7 when the catalyst was thoroughly mixed with the borohydride solution under the conditions of the experiment. In view of the lower initial temperature of the solution in Run 5, direct comparison of the data from Run 5 with those from Runs 4, 6, and 7 is not valid.

Experimental data from the four 1/10-scale runs are shown below:

	Run No.			
	8	9	10	11
Weight of NaBH_4 , lb	10.0	10.0	10.0	10.0
Weight of catalyst ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$), lb	4.0	4.0	3.0	3.2
Volume of river water, gal	56	56	56	56
Initial temperature, F	33	34	37	42
Temperature rise, F	58	58	60	59
Complete reaction time, min	74	84	112	91
Per cent reaction	100	100	100	*
Hydrogen evolved, cu ft	353	362	364	*
Water vapor evolved, cu ft	8	8	10	*

*Data being processed.

The results from Run 3 (described in our letter report dated April 14, 1959) and Runs 8 and 9 indicate that full-scale generation

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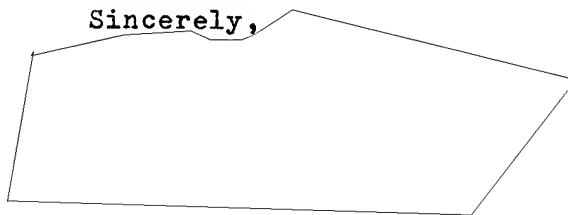
can be completed in 60 minutes or less, using 40 pounds of catalyst, only at temperatures of about 40 F or above.

A detailed check of information on the solubility of cobaltous chloride hexahydrate in various handbooks indicates that the maximum solubility of catalyst in 5 gallons of solution is approximately 37 pounds at 86 F and 30 pounds at 32 F. Tenth-scale Runs 10 and 11 were made therefore to obtain low-temperature generation times using less than 4.0 pounds of catalyst. In Run 10, ~~3~~ 3 1/2 pounds of catalyst was dissolved to provide 1/2 gallon of solution, and in Run 11, the 1/2 gallon of catalyst solution was saturated at 42 F. The results indicate that the low-temperature limit for 60-minute generation is above 42 F when saturated catalyst solution is used.

It is planned to determine the low-temperature limit of generation (time) using saturated catalyst solution in May. After this quantity is established, lower temperature generations will be conducted using more concentrated borohydride solutions.


The total appropriation under this Task Order was \$62,579. As of May 1, 1959, the unexpended balance was approximately \$8,100.

Sincerely,



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In Duplicate

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